

# Carbon Determination by Acid CO<sub>2</sub> Evolution

***This method is used for the quantitative determination of inorganic carbonate and bicarbonate (as total carbonate) by acid leaching and gravimetric determination of evolved carbon dioxide.***

## Principle of Technique

A weighed portion of sample is placed in a flask fitted with an addition funnel through which 10% HCl is added. A stream of nitrogen is passed through the solution; the resulting gases are passed through a condenser and a trap filled with Anhydrone (magnesium perchlorate) to absorb the water vapor. The gases then pass through a weighed absorption bulb containing Ascarite (sodium hydroxide on asbestos) on the bottom and more Anhydrone on top. The CO<sub>2</sub> is absorbed on the Ascarite, and the Anhydrone absorbs any water produced by the reaction. When the reaction is complete, the bulb is reweighed to determine the weight percent of acid-generated CO<sub>2</sub>.

## Samples

**Form.** Samples may be either liquid (aqueous) or solid.

**Size.** Sample size ranges from 0.025 to 0.5 g, depending on sample type and carbonate concentration.

**Preparation.** Solids must be finely ground before analysis.

## Limitations

The method is destructive. Nonaqueous liquids cannot be analyzed, and inhomogeneous materials will give variable results.

If the material does not wet well, results will be biased low. Glycerol may be added to improve wetting.

The detection limit is about 0.5 mg CO<sub>2</sub>. Depending on the sample size, this allows the detection of an analyte present at about 0.1% in a sample.

## Estimated Analysis Time

After a 2-h setup and calibration of the equipment, each measurement requires between 30 and 60 min.

## Capabilities of Related Techniques

Total carbon can be determined by an automated carbon-hydrogen-nitrogen (CHN) analyzer. Both inorganic carbon (carbonate and bicarbonate) and organic carbon can be determined by a total organic carbon (TOC) analyzer. This is the method of choice for water samples when the carbon concentration is 0.01 to 1000 ppm. Carbon at parts-per million concentrations in metals, alloys, and other materials is usually measured by other specialized equipment.

Carbonate, bicarbonate, and carbon dioxide can also be measured in water by ion-selective electrode or by titration.

## Examples of Applications

- Analysis of minerals, coal, and shale.
- Analysis of process waters, such as waters from oil shale retorting operations.

